Volatile Organic Compounds By Gas Chromatography/Mass Spectrometry (GC/MS)
EPA 8260C - 2006

Facility Name:_____ LAB ID_____

Assessor Name:_____ Analyst Name:_____ Inspection Date:_____

Records Examined: SOP Number/ Revision/ Date _____ Analyst:_____

Sample ID: _____ Date of Sample Preparation:_____ Date of Analysis:_____

Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
1. Were non-PTFE and rubber components of the GC/MS system avoided?	4.2				
2. Were analytical areas, sample storage areas, and analyst clothing isolated from sources of Methylene Chloride?	4.6				
3. Was a trip blank of organic-free reagent water carried through sampling, handling, and storage protocols to check for contamination during shipment and storage?	4.7				
4. Did the GC oven temperature program include a post-analysis bake out period to ensure that semi-volatile hydrocarbons were volatilized?	4.10				
5. Were stock standards stored w/minimal headspace in bottles w/ PTFE-lined screw caps at ≤ 6° C and protected from light?	7.74				
6. Were fresh standards prepared if checks against the initial calibration exceeded 20% drift?	7.7.5				
7. Were secondary dilution standards stored in vials with no headspace and replaced after 2-4 weeks unless their continued acceptability could be documented?	7.8				
8. Were secondary dilution standards containing gaseous compounds replaced after 1week unless their continued acceptability could be documented?	7.8				
9. Was each sample spiked with the surrogate spiking solution prior to analysis?	7.9				
10. Was the appropriate amount of internal standard added to each sample and calibration standard?	7.10 7.12.4				
11. Were the area counts of the internal standard peaks between 50% - 200% of the areas of the target analytes in the mid-point calibration analysis?	7.11				
12. Were initial calibration standards prepared from fresh stock standards and secondary dilution standards at a minimum of five different concentrations?	7.12.1 11.3.2				

Notes/Comments

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13. Were calibration verification standards prepared at a concentration near the mid-point of the initial calibration range?	7.12.2				
14. Were all target analytes for the analysis included in the initial calibration and calibration verification standards?	7.12.13				
15. Were quantitative results never reported for target analytes that were not included in the initial calibration?	7.12.13				
16. Were the LCS and matrix spike prepared from the same source material as the initial calibration standards?	7.13				
17. Were calibration standards stored w/minimal headspace in amber vials w/ PTFE-lined caps at ≤ 6° C and protected from light?	7.14				
18. Were aqueous samples stored with minimal or no headspace?	8.2				
19. Were samples stored separately from standards and from other samples expected to contain significantly different concentrations of volatile compounds?	8.3				
20. Were storage blanks used to monitor potential cross-contamination due to improper storage conditions?	8.3 NOTE				
21. Was the GC/MS tuned, on injection of 50 ng or less of BFB, to meet the BFB acceptance criteria prior to calibration and for each 12 h analysis period?	9.2 11.3.1				
22. Did all subsequent standards, samples, MS/MSDs, LCSs, and blanks associated with a BFB analysis use identical instrument conditions?	11.3.1.2 NOTE				
23. Did the GC/MS system meet calibration acceptance criteria every 12 h during analysis?	9.2				
24. Did the retention time of each sample component fall within the retention time widow of the corresponding standard component?	9.2				
25. If sample dilutions were performed using an autosampler, had the laboratory verified that the accuracy of those dilutions were equivalent to the accuracy achieved by an experienced analyst performing manual dilutions?	9.3 11.5.6.5				
26. Were method blanks, carried through all sample processing steps, analyzed prior to each set of samples, every 12 h during analysis, and each time there was a	9.4 9.5.4 11.4.4				

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change in reagents?					
27. Was at least one matrix spike analyzed with each batch of samples?	9.5.1				
28. Was at least one sample duplicate or matrix spike duplicate analyzed with each batch of samples? NOTE: *if samples are not expected to contain target analytes, laboratories should use a MS/MSD pair.*	9.5.1				
29. Were surrogate recovery data from individual samples evaluated against the control limits developed by the laboratory?	9.6				
30. Were all internal standards and surrogates added to the samples prior to introduction to the GC/MS system?	11.1 11.5.8				
31. Were all matrix spiking compounds added to the applicable samples prior to introduction to the GC/MS system?	11.1				
32. Was direct injection of aqueous samples used only for determination of volatiles at the toxicity characteristic level of at concentrations exceeding 10,000 µg/L?	11.1.1				
33. Was the response factor calculated for each analyte relative to the nearest internal standard?	11.3.4				
34. Were the mean response factors and the relative standard deviations (RSD) of the response factors calculated for each target analyte?	11.3.4.1				
35. If more than 10% of target analytes exceeded 20% RSD and failed to meet a correlation coefficient of 0.99, was corrective action taken and the system recalibrated prior to analyzing samples?	11.3.4.2				
36. Were the mean and relative standard deviation (RSD) of the response factors calculated for each target analyte?	11.3.5				
37. If the RSD>20%, was the average response factor (RF) not used to calculate results unless the concentration was reported as an estimate?	11.3.6.1				
38. Was BFB ≤ 50 ng analyzed, and acceptance criteria met, prior to calibration and every 12 h during sample analysis?	11.4 11.4.1				
39. Was calibration verified immediately following initial calibration and every 12 h during sample analysis using a second source standard?	11.4 11.4.2				

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40. Was a calibration verification (CCV) standard analyzed, and acceptance criteria met, prior to sample analysis and every 12 h during sample analysis?	11.4 11.4.3 11.4.5.1				
41. If minimum acceptance criteria were not met, was the system evaluated and corrective action taken before beginning sample analyses?	11.4.5.2				
42. If linear regression analysis was used for quantitation of data, was the lowest calibration point recalculated as if it were an unknown sample and found to be within ±30% of its known concentration?	11.4.5.6				
43. Were the retention times of the internal standards in the CCVs monitored against the retention times of the same compounds in the mid-point standard of the most recent initial calibration?	11.4.6				
44. Was corrective action taken if the retention time of any internal standard differed by more than 10 seconds from the retention time of the initial calibration mid-point?	11.4.6				
45. Were the area counts of internal standards in the CCVs monitored against the area counts of the same compounds in the mid-point standard of the most recent initial calibration?	11.4.7				
46. Was corrective action taken if the area count of any internal standard differed by more than a factor of two (-50% to +100%) from the retention time of the initial calibration mid-point?	11.4.7				
47. Were all samples and standards allowed to come to ambient temperature before analysis?	11.5.3				
48. If it was necessary to split the sample from a single vial into two aliquots for analysis, were both aliquots prepared at the same time?	11.5.4				
49. If analysis of both aliquots of a split sample was required, was the second aliquot analyzed within 24 hours?	11.5.4				
50. Were samples to be composited cooled to <6° C to minimize volatilization of analytes?	11.5.7.1.1				
51. Were samples that exceeded the initial calibration range diluted and reanalyzed?	11.5.11				
52. When ions from a sample saturated the detector, were reagent water blanks analyzed to determine whether there was a need for decontamination?	11.5.11.1				

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53. Did all dilutions maintain response of major constituents in the upper half of the linear range of the curve?	11.5.11.2				
Qualitative Analysis					
54. Were the relative retention times(RRTs) of sample components within ±0.06 RRT units of the RRTs on the standard component?	11.6.1.2				
55. Did the relative intensities of characteristic ions agree within 30% of these ions in the reference spectra?	11.6.1.3				
56. When structural isomers were identified individually, did they have GC resolution such that the heights of their valleys were less than 50% of the average of the two peak heights?	11.6.1.4				
57. When structural isomers were not sufficiently resolved, were they identified as isomeric pairs?	11.6.1.4				
Quantitative Analysis					
58. Were quantitations of compounds based on the internal standards with retention times nearest to those analytes?	11.7.1				
59. When structural isomers were identified individually, did they have GC resolution such that the heights of their valleys were less than 50% of the average of the two peak heights?	11.7.4				
60. Was the resolution verified on the mid-point concentration of the initial calibration as well as the CCV?	11.7.4				
Quality Control from EPA 8000C					
61. Was the instrument performance checked every 12-hour analysis period according to some sort of QC program?	8000C 9.2.1				
62. When calibration verification acceptance criteria could not be achieved, was the instrument recalibrated?	8000C 9.2.5				
63. Were method blanks analyzed prior to analyzing samples?	8000C 9.2.6				
64. Were method blanks prepared at a frequency of 5% or every 20 samples?	8000C 9.2.6.1 8260B 8.4				

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65. Were method blanks and LFMs subjected to the same procedures as samples?	8000C 9.2.6.4 9.7.8				
66. Were method blanks and LFMs subjected to the same procedures as samples?	8000C 9.2.6.4 9.7.8				
67. Were method blanks determined to be lower in analyte concentration than some target dictated by a QC program?	8000C 9.2.6.5				
68. Were r^2s/correlation coefficients/coefficients of determinations of calibration curves all ≥0.99 or the RSDs of calibration analytes ≤20%?	8000C 9.3.2 11.5.2				
69. Were calibration verifications within ±20% of the responses calculated during initial calibrations?	8000C 9.3.6 11.5.1				
70. When the facility or new analysts began this method or there were significant changes in instrumentation were IDC's performed?	8000C 9.4.1				
71. Did IDC's consist of the mean recoveries of at least four standards falling within 70% to 130% and the calculation of standard deviations?	8000C 9.4				
72. Was a LFM/LFMD pair or Sample/Duplicate/LFM set prepared and analyzed with each batch of up to 20 samples?	8000C 9.5. 8260B 8.4				
73. Were LFMs and surrogate recovery values within 3 standard deviations of their average percent recoveries?	8000C 9.7.3				
74. Were LCS samples consisting of reagent matrix spiked to the same concentration as LFMs prepared and analyzed with each batch? (**Not** second-source)	8000C 9.5. 8260B 8.4				
75. Were failed data included in Control Limit calculations to avoid "censored data sets?"	8000C 9.7.8				

Notes/Comments